

# TEST PROCEDURE

**TP 108B**

<b>Title</b> Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends	<b>Page Number</b> 1 of 28
<b>Originator</b> Carl Scarbro	<b>Supersedes</b> TP 108A
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## Implementation Approval

Original Test Procedure Authorized by EPCN #90 on 10-15-90

## Revision Description

- (1) 09-30-94 The purpose of this change is to revise the procedure as described in EPCN #170.

**Note:** Specific brand names in EPA/EOD procedures are for reference only and are not an endorsement of those products.

Table of Contents

1. Purpose ..... 3

2. Test Article Description ..... 3

3. References ..... 3

4. Required Equipment ..... 4

5. Precautions ..... 6

6. Visual Inspection ..... 6

7. Test Article Preparation ..... 7

8. Test Procedure ..... 11

9. Data Input ..... 14

10. Data Analysis ..... 14

11. Data Output ..... 14

12. Acceptance Criteria ..... 15

13. Quality Control Provisions ..... 17

Attachments

Attachment A ..... 19

Attachment B ..... 20

Attachment C ..... 21

Attachment D ..... 22

Attachment E ..... 23

Attachment F ..... 24

Attachment G ..... 25

Attachment H ..... 26

Attachment I ..... 27

Attachment J ..... 28

## 1. Purpose

The purpose of this procedure is to determine the vapor pressure of gasoline and gasoline-oxygenate blends using the Herzog digital vapor pressure analyzer.

This procedure deviates from 40 CFR Part 80, Appendix E, Tests for Determining Reid Vapor Pressure (RVP) of Gasoline and Gasoline-Oxygenate Blends [54 FR 11868 (March 22, 1989) and 55 FR 25833 (June 25, 1990)] in the number of significant figures used to report the test results.

## 2. Test Article Description

A 1-quart (1-liter) sample container that is 70-85 volume percent filled with a gasoline or gasoline oxygenate blend.

## 3. References

- 3.1 40 CFR Part 80, Appendix E, Tests for Determining Reid Vapor Pressure (RVP) of Gasoline and Gasoline-Oxygenate Blends [54 FR 11868 (March 22, 1989) and 55 FR 25833 (June 25, 1990)]
- 3.2 "1989 Annual Book of American Society for Testing and Materials (ASTM) Standards;" Section 5; Petroleum Products, Lubricants, and Fossil Fuels
  - 3.2.1 D 4057 "Practice for Manual Sampling of Petroleum and Petroleum Products"
  - 3.2.2 E 1 Specification for ASTM Thermometers
  - 3.2.3 D 4953 Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends
- 3.3 "Operating Instructions for Vapor Pressure Reid Method," Herzog operating manual
- 3.4 "Handbook of Chemistry and Physics," Section D 217, 65th Edition
- 3.5 "Reference Data for Hydrocarbons and Petrosulfur Compounds," Bulletin No. 251, Phillips Chemical Company

#### 4. Required Equipment

4.1 Herzog semi-automatic vapor pressure analyzer. Refer to 40 CFR Part 80, Appendix E, Annex A1; and Method 2-Herzog Semi-Automatic Method, Step 4.2, for specific description of the following equipment:

- 4.1.1 Fuel chamber
- 4.1.2 Air chamber
- 4.1.3 Transfer apparatus
- 4.1.4 Mercury manometer
- 4.1.5 Water bath
- 4.1.6 Cooling bath

4.2 Reagents:

- 4.2.1 n-pentane (pure grade and commercial)
- 4.2.2 3,3-dimethylbutene-1 (pure grade)
- 4.2.3 2,2,4-trimethylpentane (iso-octane) (pure grade)
- 4.2.4 2,2-dimethylbutane (neohexane) (pure grade)
- 4.2.5 3-methylpentane (pure grade)

4.3 Documentation:

- 4.3.1 Form 108-01, RVP Analysis Label (Attachment A)
- 4.3.2 Form 108-02, Vapor Pressure Quality Control Chart (Attachment B)
- 4.3.3 Form 108-03, Herzog Calibration Log (Attachment C)
- 4.3.4 Form 3500-5, EPA Fuels Field Inspection (Attachment D)

- 4.3.5 EPA Form 3700-2, Analysis of Lead in Gasoline (Attachment E)
- 4.3.6 Target Pressures on Stabilized Air Chambers (Attachment F)
- 4.3.7 Precision Data Report - Analysis of Laboratory Duplicates (Attachment G)
- 4.3.8 Accuracy Data Report- Analysis of Laboratory Standards (Attachment H)
- 4.3.9 Vapor Pressure Statistical Summary (Attachment I)
- 4.3.10 Vapor Pressure Precision and Accuracy Calculation Formulas (Attachment J)
- 4.4 Other Equipment
  - 4.4.1 Refrigerator
  - 4.4.2 Ice machine
  - 4.4.3 Coolers
  - 4.4.4 Compressed air/vacuum manifold
  - 4.4.5 Compressed air and lab vacuum
  - 4.4.6 Environmental Protection Agency (EPA)-approved fume hood
  - 4.4.7 8-ounce clear PVC coated bottles
  - 4.4.8 00 rubber stoppers
  - 4.4.9 Lab Coat
  - 4.4.10 Safety Glasses
  - 4.4.11 Wiper Cloths
  - 4.4.12 Rubber Gloves
  - 4.4.13 Waste Can with Fuel Chamber Key

## 5. Precautions

- 5.1 Gasoline is flammable and toxic. The test assembly with gasoline is under pressure and can spray the analyst when it is disassembled for cleaning. Analysts must perform all phases of the procedure where the sample container is open under an EPA-approved fume hood. Safety glasses and gloves are required.

Samples must be kept in laboratory areas with single-pass air handling and an automatic fire suppression system. Fume hoods must be cleared of sample and waste products at the end of the work day.

- 5.2 Sample in accordance with 40 CFR Part 80, Appendix D.
- 5.3 All vapor pressure tests will be the first type of analysis performed on a sample. Multiple vapor pressure analyses may be performed on any sample. The order of testing must be recorded.
- 5.4 The transfer apparatus, fuel chamber, and sample must be chilled to 32-34 °F before starting the test procedure. In all cases, cool the sample container and contents to 32-34 °F before opening the container.
- To ensure that there is sufficient time to reach this temperature, directly measure the temperature of a similar liquid, in a similar container at a similar temperature, and place it in the ice bath at the same time as the sample.
- 5.5 Once the test procedure begins, the analyst should proceed with the objective of minimizing the warming of the sample and the chilled hardware before the liquid and vapor chambers are assembled.

## 6. Visual Inspection

- 6.1 Samples in leaking containers should have Comment Code 1 marked on Form 108-01 (Attachment A). Discoloration of any seals placed over the cap or the presence of gasoline vapors in the shipping container indicates a leak.
- 6.2 Samples in glass containers must be observed for phase separation and clarity after they are cooled to 32-34 °F and before analysis. Samples in metal cans must be observed for those properties after analysis (see Step 109).

For samples in glass containers, shake the container for 5 seconds. Observe the sample for clarity and phase separation.

If the sample is cloudy, mark Comment Code 2 on Form 108-01. If the sample separates into two or more phases, mark Comment Code 3 on Form 108-01.

- 6.3 The Herzog water bath must be filled between the level marks on the Herzog thermometer well.
- 6.4 The ice bath should be a slurry of crushed ice and water. The bath's depth should be such that the surface of the slurry is at or above the surface of the sample in the container.

## 7. Test Article Preparation

- 7.1 All samples found to be in violation of the applicable enforcement standard must be analyzed as laboratory duplicates.

Additional samples may be picked at random to be analyzed as laboratory duplicates so that at least 1 sample in 20 is duplicated.

The determination of the transducers to be used, the calculation procedure, and the tolerance to be applied are in Attachment B, Vapor Pressure Quality Control Chart.

The initial analysis will be subtracted from the duplicate. The result is recorded on the control chart and compared with the 95% confidence limits.

Corrective action for an out-of-limits lab duplicate analysis is outlined in Step 12.6.

- 7.2 A quality control standard is to be analyzed daily as outlined in Attachment B and defined in Step 12.6. The listed Average Pressure for the prescribed control standard will be subtracted from the observed value and the result recorded on Attachment B, Vapor Pressure Quality Control Chart.

Corrective action for an out-of-limits measurement is described in Step 12.7.

- 7.3 Transducers are calibrated against a mercury manometer at the beginning of each test day and when they differ from 0.00 psi at ambient pressure by  $\pm 0.03$  psi.

The transducer is adjusted to read 0.00 psi at atmospheric pressure and the same pressure as the manometer at an applied pressure of 10 psi-15 psi.

Calibrated transducers will have no correction factor applied to subsequent analyses. The daily transducer calibration is recorded on the Herzog Calibration Log (Attachment C).

- 7.4 The water bath of the Herzog instrument must be at  $100\text{ }^{\circ}\text{F} \pm 0.2\text{ }^{\circ}\text{F}$ . The measuring device must be an ASTM Reid Vapor Pressure Thermometer  $18\text{ }^{\circ}\text{F}$  having a range from  $94\text{-}108\text{ }^{\circ}\text{F}$  and conforming to the requirements in Specification E1.

The thermometer will be located in the Herzog thermometer well.

- 7.5 Document Preparation:

- 7.5.1 Affix a blank copy of Form 108-01 (Attachment A) to EPA Form 3500-5 (Attachment D) upon removing the sample bottles and Form 3500-5 from the shipper.

Compare the following data on Form 3500-5 with the data on the sample bottle seal, EPA Form 7500-2 (Attachment E).

Sample #

Sample Date

Inspector

- 7.5.2 If a conflict exists between any of the above data found on Form 3500-5 and the sample seal, Form 7500-2, notify the supervisor immediately and he/she will instruct you which form will be edited (in general, the data on the bottle seal takes precedence).

The analyst will edit the conflicting data by striking a single horizontal black ink line through the incorrect data and clearly printing the correct information, his/her initials, and the date as close to the incorrect data as possible (see Attachment D).

- 7.6 Prior to the initial test, filling of the sample container is verified as follows:

- 7.6.1 With the sample at a temperature of  $32\text{-}34\text{ }^{\circ}\text{F}$ , take the container from the ice bath, wipe it dry with an absorbent material, and measure its ullage. The sample content, as determined by a gauge designed for the specific container, shall equal 70%-85% of the container capacity.

- 7.6.2 If the sample container is less than 70% full, mark Comment Code 4 on Form 108-01. If it is greater than 85% full, mark Comment Code 5 on Form 108-01.

7.6.3 If the container is more than 80% full, pour out enough sample to bring the container contents within the 70%-80% range. Under no circumstances may any sample poured out be returned to the container.

If the sample is less than 70% full after the above operation, mark Comment Code 6 on Form 108-01.

**Note:** The sample must be 70-85% of container capacity when received and it must be within 70-80% to complete this test.

7.7 Air saturation of the sample in the container is as follows:

7.7.1 With the sample at a temperature of 32-34 °F, take the container from the ice bath; wipe it dry with an absorbent material; unseal it for several seconds, taking care that no water enters the sample; re-seal it; and shake it vigorously for 5 seconds.

Return it to the bath for a minimum of 2 minutes.

7.7.2 Repeat Step 7.7.1 twice for a total of three air saturations. Return the sample to the ice bath for at least two minutes before beginning the analysis.

7.8 Cleaning of the air chamber, fuel chamber, T-handled coupler, and transfer apparatus is as follows:

7.8.1 Disconnect the quick action T-handled coupler. Separate the air and gasoline chambers and discard any remaining sample.

Rinse the chambers and the transfer connections in a manner such that all the inner surfaces are wetted with commercial grade n-pentane. Drain the rinse into the waste can with the fuel chamber key.

Dry the interior surfaces of the chambers with dried and filtered compressed air or by connecting them to a vacuum source. Dry the T-handled fitting and spiral tubing by connecting them to a vacuum source or by blowing dried and filtered compressed air through them.

Use the gasoline chamber as the air chamber stoppering device to prevent water from entering the chamber during its equilibration to 100 °F ±0.2 °F for the next test.

7.8.2 Rinse the interior surfaces of the transfer apparatus with commercial grade n-pentane. Dry the interior by blowing dried and filtered compressed air or by connecting to a vacuum source.

7.9 Preparation of the air chamber is as follows:

7.9.1 Inspect the teflon washers at both ends of the air chamber. They must be free of dirt or other particles. If they are not, clean them with a cotton swab.

If they can not be cleaned, replace them with new ones.

7.9.2 Screw the T-handled fitting into the smaller opening in the air chamber. Seal the larger opening with a clean and dry fuel chamber.

7.9.3 Check the transducer display for transducer drift. It must read  $0.00 \pm 0.03$  psi.

If it does not, calibrate the transducer as described in Step 7.3.

7.9.4 Connect the assembled chambers to the transducer with the quick disconnect fitting on the T-handled fitting. Observe the air temperature from where the chambers were stored before assembly.

Immerse the assembly to its test position in the  $100 \text{ }^\circ\text{F} \pm 0.2 \text{ }^\circ\text{F}$  water bath for not less than 10 minutes just prior to coupling it with a sample-filled fuel chamber.

The indicated pressure should stabilize according to the above air temperature and to the values stated in Attachment F within that time period.

If the pressure is below the lower value, check for leaks in the chamber or T-handled fitting.

If the pressure is high, the chambers may be contaminated and must be cleaned again.

If the pressure is within specification, do not remove the chambers from the water bath until the fuel chamber has been filled with sample as described in Step 101.

7.10 To prepare the fuel chamber, seal, the inlet of a fuel chamber that has successfully passed the pressure check in Step 7.9.4 with a 00 stopper and place the chamber in the cooling bath for at least 10 minutes.

Keep the fuel chamber upright and do not immerse it over the top of the coupling threads.

- 7.11 The transfer apparatus is cooled to 32-34 °F by placing it in a cool air bath at 32-34 °F for at least 10 minutes.

## 8. Test Procedure

- 101 To transfer the sample, remove the chilled sample container from the ice bath, dry the exterior of the container with an absorbent material, uncap the container, and insert the chilled transfer apparatus.
- 102 Remove the fuel chamber from the ice bath, dry it with an absorbent material, and invert it over the shorter tube of the transfer apparatus.
- 103 Invert the entire system rapidly so that the fuel chamber is upright, its bottom is engaged with the chamber key in the gasoline waste can, and the end of the transfer apparatus touches the bottom of the fuel chamber.
- 104 Fill the fuel chamber so the sample overflows the chamber and drains into the waste can. Withdraw the transfer apparatus from the chamber, while allowing the sample to continue flowing up to the moment of complete withdrawal.
- 105 To assemble the apparatus, disconnect an air chamber meeting the conditions of Step 7.9.4 from its transducer. Remove the air chamber from the water bath and quickly dry the exterior of the chamber with an absorbent material, giving particular attention to the connection between the air chamber and the fuel chamber.
- Remove the chamber that was used to seal the air chamber (this fuel chamber may now be placed in the ice bath to be used in subsequent tests).
- As quickly as possible, without spilling the sample, couple the air chamber to the filled fuel chamber. The assembly must be done without undue movement, which could promote exchange of room temperature air with the 100 °F air in the air chamber.
- Not more than 10 seconds shall be consumed in coupling the two chambers.
- 106 To introduce the apparatus into the bath, hold the apparatus vertically, air chamber end up, and immediately reconnect the spiral tubing to the transducer with the quick disconnect fitting.
- Rotate the apparatus 110-120 °F, air chamber end down, for 5 seconds to allow sample to flow into the air chamber without getting into the spiral tube.

Increase the angle of the tilt vertically with the air chamber end down and hold until the pressure increases slowly (approximately 0.01 psi per second). This takes about 20 seconds.

Place the assembled apparatus into the 100 °F  $\pm$ 0.2 °F water bath so that the bottom of the fuel chamber engages the drive coupling and the other end rests on the support bearing.

107 Record on Form 108-01 the transducer number, the time of day, the analysis date, and the analyst's initials. Return the re-sealed sample to the ice bath.

108 Observe the apparatus for leaks throughout the test. Vapor and liquid leaks under the surface of the water bath will be continuous and visually evident.

Leaks in the air chamber to the transducer connector can be detected through any decrease of measured vapor pressure. In case of any leaks, record the highest vapor pressure reading as the sample's Reid Vapor Pressure and mark Comment Code 7 on Form 108-01.

Tag all associated chambers, fittings, and transducers as suspect, and immediately notify the supervisor of possible equipment problems.

109 If the sample is in a metal can and no further analyses are to be performed on the sample, check the remaining sample for phase separation. This is done by shaking the container for 5 seconds and pouring a quantity of the sample into a clear glass container.

Observe it as in Step 6.2.

If the sample is cloudy, mark Comment Code 2 on Form 108-01. If the sample separates into two or more phases, mark Comment Code 3 on Form 108-01.

110 To measure the vapor pressure, observe and record the transducer reading to the nearest 0.01 psi after the assembled apparatus has been in the bath for at least 5 minutes.

At intervals of not less than 2 minutes, observe and record pressure readings until the last consecutive transducer readings are within 0.01 psi of each other. The last reading is the Reid Vapor Pressure.

On EPA Form 3500-5, record this value to the nearest 0.01 psi as the Reid Vapor Pressure of the sample.

- 111 To verify the integrity of the sample contained in the assembled apparatus, perform the following operations immediately after removing the assembly from the water bath: Dry the exterior surface with absorbent material and, with the assembly in an upright position, vent the pressure by removing the T-handled fitting and spiral tube.
- Caution: not venting in this manner may result in the dangerous spraying of gasoline through the vapor chamber pressure relief vent onto the operator and into the room.
- Disconnect the air chamber from the fuel chamber. Drain the contents of the fuel and air chambers into a dry, 8-ounce clear glass bottle. Seal the container and shake it vigorously for 5 seconds.
- Determine whether the sample is clear and bright and free of a second phase.
- If it is not, immerse the bottle in the 100 °F water bath to about 1 inch above the top of the sample level for at least 15 minutes in order to heat the sample to test temperature.
- Remove the sample from the water bath, immediately shake it vigorously for 5 seconds, and observe the sample.
- If the sample is not clear and bright and free of a second phase, mark Comment Code 8 on Form 108-01.
- 112 Compare the RVP standard on EPA Form 3500-5 with the measured pressure of the sample(s) identified on the form.
- If the measured vapor pressure exceeds that value, the sample must be analyzed again. Start with a chilled sample (32-34 °F) and proceed to Section 8, Step 101.
- A tag must be tied to the sample. That tag will have written on it: the sample number, the Field Operations Support Division (FOSD) office responsible for the area from where the sample was taken, the date it was received, and the date it was analyzed for vapor pressure.
- 113 Review EPA Form 3500-5 for any further analyses requested by FOSD for the sample(s) in question.
- If further analyses are requested, a tag is tied to the sample. The tag will have written on it: the sample number, the FOSD office responsible for the area from where the sample was taken, the date it was received, the date it was analyzed for vapor pressure, and the other analyses requested.
- Custody of the sample will be given over to the analyst responsible.

**9. Data Input**

9.1 Form(s) 108-01 must be filled out in full and affixed to EPA Form 3500-5. These documents will be used to generate FOSD and Fuels and Chemistry Service (F&CS) reports on the analyses of the individual samples.

9.2 Form 108-03, Herzog Calibration Log, and Form 108-02, Vapor Pressure Quality Control Chart, are stored in log books in the Physical Chemistry Laboratory.

They document F&CS's conformance to this procedure and are used by F&CS to generate its monthly test and periodic quality control reports.

**10. Data Analysis**

10.1 The analyst must verify the completeness and accuracy of Form 108-01; EPA Form 3500-5; Form 108-03, Herzog Calibration Log; and Form 108-02, Vapor Pressure Quality Control Chart .

All corrections to these forms must be dated and initialed.

10.2 The duplicate and standard data on the above forms will be used to calculate precision and accuracy.

**11. Data Output**

The completed EPA Forms 3500-5 and Form 108-01 will be photocopied. The originals will be given to FOSD's data staff.

They will enter the data into their data handling system to produce reports for their needs. They will retain the original forms and use them as evidence in case of litigation.

F&CS will use the copies to produce the monthly Results of Gasoline Analyses report and the Precision and Accuracy of Vapor Pressure Testing report (see examples at Attachments G through J).

**12. Acceptance Criteria**

**Note:** Steps 12.6 through 12.8 are included specifically to define data quality as required by EPA Order 5360.1, Policy and Program Requirements to Implement the Quality Assurance Program. They should not be interpreted as a change in EPA's policy regarding enforcement tolerance as set forth in 54 Federal Register 11877 (March 22, 1989).

12.1 The sample container did not leak.

12.2 The sample container was 70%-85% full.

12.3 The sample, as received, and the analysis sample did not phase separate and were not cloudy.

12.4 The apparatus did not leak.

12.5 Duplicate analyses were done at the frequency stated in Step 7.1 and met the following criteria:

12.5.1 The difference between successive test results obtained under constant operating conditions and identical test materials would, in the normal and correct operation of the test method, exceed the following value only in 1 case in 20.

The field duplicate precision data was based on a 1988 ASTM cooperative test program. The laboratory duplicate precision data was based on the 1989 EPA Enforcement Program.

<u>Sample Type</u>	<u>Container Volume</u>	<u>Container Type</u>	<u>95 % Tolerance (psi)</u>
Field Duplicate	1 quart	glass	±0.26
Lab Duplicate	1 quart	glass	+0.20 to -0.22

12.5.2 When the absolute difference between field duplicates exceeds the applicable tolerance, analyze the samples again using the same transducers. However, each sample will be tested with the transducer that was not used for the original test.

If the resulting pair of lab duplicates and the field duplicates are within their respective tolerances, the initial analyses are considered the 1-in-20 out-of-tolerance analyses which are expected for this process. Continue the analyses.

If both sets of field duplicate analyses are out of tolerance and both sets of lab duplicates are within tolerance, a sampling or sample handling error may have occurred.

If possible, new samples should be taken and analyzed.

If either of the lab duplicates are out of tolerance, the process is considered out of tolerance; proceed to Step 12.5.3.

- 12.5.3 When the absolute difference between laboratory duplicates exceeds the applicable tolerance, the analyst must examine the transducer calibration, check the test assemblies for leaks, and notify the supervisor.

If the equipment is at fault, the process is considered out of tolerance. Proceed to Step 12.7.

If the transducers are calibrated correctly and there are no system leaks, the sample will be analyzed again.

If the second test set is within tolerance, the initial set is considered the 1-in-20 analyses out of tolerance which is expected for this process.

If not, the process is out of tolerance; proceed to Step 12.7. All duplicate data will be recorded and reported for the monthly quality assurance report and on the Vapor Pressure Quality Control Chart.

- 12.6 Analysis of standards must meet the following criteria:

- 12.6.1 A quality control standard must be analyzed once a day for each instrument in use that day. The QC standard and transducer used are identified in the Vapor Pressure Quality Control Chart (see example, Attachment B).

If the measured vapor pressure of a standard exceeds the stated tolerance, analyze the next standard in the schedule (see example, Attachment B).

If that standard is in conformance with the acceptance criteria, the initial analysis is considered the 1-in-20 out-of-tolerance measurement expected for this process; continue to analyze samples.

If not, the process is out of tolerance; proceed to Step 12.7.

- 12.6.2 EOD has established a bias statement for the following four pure hydrocarbon compounds. This data was based on analyses done during EPA's 1989 Volatility Enforcement Program except for iso-octane, which was done at EPA prior to that date.

Dry Reid Vapor Pressure (psi)

<u>Compound</u>	<u>Published</u>		<u>Mean</u>	<u>95% Tolerance</u>	
	<u>VP (psi)</u>	<u>n</u>		<u>Lower</u>	<u>Upper</u>
iso-octane	1.71	9	2.02	1.84	2.20
3-methylpentane	6.10	19	6.23	6.05	6.42
2,2-dimethylbutane	9.86	26	9.89	9.70	10.08
n-pentane	15.57	20	15.38	15.16	15.60

- 12.7 The analyst, in conjunction with the supervisor, must attempt to identify the source of the out-of-tolerance situation. Corrective action will be taken to ensure that further analyses meet the specifications of this procedure. They must also identify what analyses may be affected by the out-of-tolerance situation.

The above information must be written on a Discrepant Test Review Form 106-01 and submitted for review, along with a copy of the applicable Vapor Pressure Quality Control Chart (Attachment B), to Quality Control.

Disposition of the sample measurements in question will be determined jointly by Quality Control, the analyst, and the supervisor.

- 12.8 Values found in the tables in Steps 12.5.1 and 12.6.2 will be updated every November to reflect the changes in precision and accuracy measured from the preceding year.

### 13. Quality Control Provisions

- 13.1 Daily transducer calibration.
- 13.2 Visual inspection for leaks, phase separation, and minimum and maximum volume limits of the sample in the sample container.

- 13.3 Pretesting of the vapor and liquid chambers for contamination and leaks.
- 13.4 Samples found to be in violation of the RVP standard on EPA Form 3500-5 are analyzed as laboratory duplicates. At least 1 sample in 20 must be a laboratory duplicate.
- 13.5 Analysis of one standard sample per instrument per day.
- 13.6 Adherence to the Vapor Pressure Quality Control Chart (Attachment B) for the analyses of duplicates and standards.

**Attachment A**

**RVP Analysis Label (Form 108-01) and a definition of Comment Codes**

Sample ID	Date	Time	Analyst	Comment Codes	T	I

T = Transducer 1, 2, or 3; I = Distress 1 or 2

Form 108-01 9/8/90

Signatures: \_\_\_\_\_ Date: \_\_\_\_\_

**Comment Codes:**

1. There is evidence that the sample container leaked.
2. The sample upon chilling to 32-34°F is cloudy.
3. The sample upon chilling to 32-34°F separates into two or more phases.
4. The received sample container upon chilling to 32-34°F is less than 70 % full.
5. The received sample container upon chilling to 32-34°F is greater than 85% full.
6. The received sample container upon chilling to 32-34°F and subsequent removal of sample in excess of 80% is below 70 %.
7. The test apparatus leaked during the analysis of the sample.
8. The analysis sample is cloudy or has two phases.

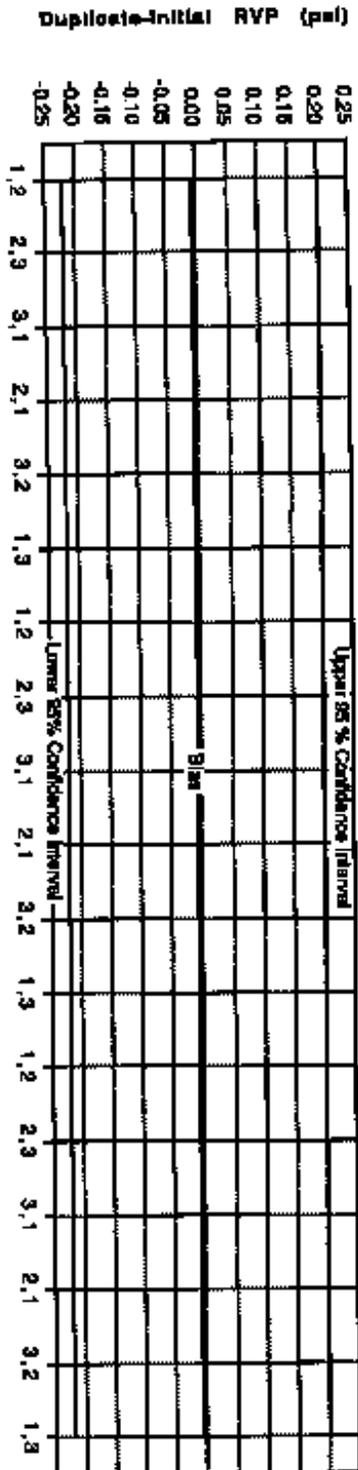
### Vapor Pressure Quality Control Chart

Attachment B

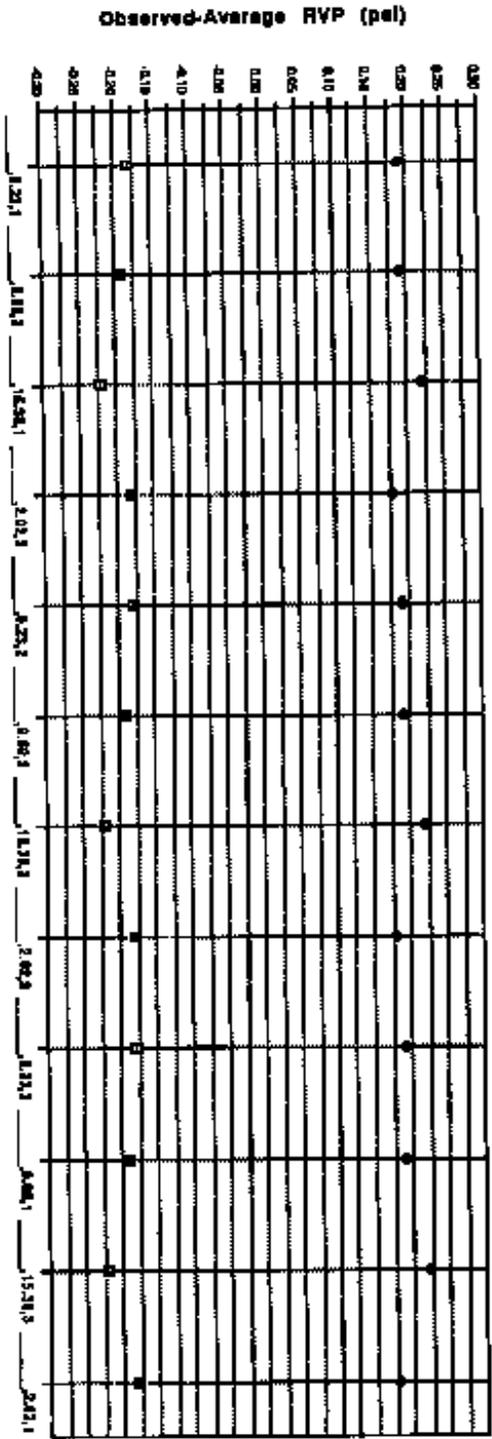
Please mark all data points with a red X

Instrument Number: \_\_\_\_\_  
Start Date: \_\_\_\_\_

#### Duplicate Control Chart



#### Initial, Duplicate Transducer, and Date Standard Control Chart



Date, Standard, Transducer

Form 109-02 9/8/90



Attachment D

**SEPA** U.S. Environmental Protection Agency  
Washington, DC 20460

## Fuels Field Inspection

Check box if you apply below  
 Inspector Class  
Inspection Number: **1010052**

Inspection Date (mm-dd-yy): **04-03-90** Time (hh:mm): **13:25** Time (hh:mm): **14:13** Type of Inspection:  Routine  Directed

Facility Type (mark only one):  Refiner  Importer  Distributor  Chemical Producer  Reseller  Carrier  Retailer  Wholesale P-C

Equipment ID# \_\_\_\_\_ Mobile Usage \_\_\_\_\_ Fuel Sampling Site \_\_\_\_\_ Volatility Sampling Instrument \_\_\_\_\_ Field Tests Conducted by \_\_\_\_\_

**1. Facility Information**

Facility Name: **Y-Pay-Move** Company, Trade or Other Name: **USEPA Gas** District Telephone No. (inc. area code): **303 450-4209**

Address: **1201 Adams Ave** City: **Ann Arbor** State: **MI** ZIP: **48103**

County: **Washtenaw** Facility Owner: **Lula Kottler** Labeled (if boxed) \_\_\_\_\_

Company Contact: **Lula Kottler** Owner: **Bud Scarbro** Analyst: **Student**

**2. Fuel Descriptions and Use Information**

Grade	Tank #	Pump #/RCH	Pump Octane	BSP Grav		Full Burn		Blended Lix		4. Test Results				
				Rate	Per	Rate	Per	Sample Date	Analysis Unit	Temp #	Test Type	Test #	Test Result	
1	1		90		49:6			4/90	5/90	MI	1	12	1	10.31
2	2		91		1:12:6			4/90	5/90	MI	2	12	1	10.32
3	3	10.0	93		1:30:6			4/90	5/90	MI	3	12	1	10.94
											3	11	1	8.0
											4	60	1	10.82
											5	60	1	10.59
											6	60	1	11.31
											4	60	2	10.53
											5	60	2	10.62
											6	60	2	11.27
											6	50	1	7.6

**3. Sample Information and Field Violations**

Temp #	Grade	Tank #	Pump #	Sample Size	Sample Type	Flash Volume	Pump, Tank or Vehicle #/V	Violations Code
1	1	1	37	7		12.2	<b>RVP STANDARD 10.5</b>	
2	2	2	313	7		9.6	<b>CHAIN OF CUSTODY RECEIVED</b>	
3	3	3	409	7		9.3	Sample numbers: <b>1010052-4,5,6</b>	
4	1	1	37	7		12.2	Date shipped to lab: <b>4/3/90</b>	
4	2	2	313	7		9.6	Seal cost: <b>6.00</b>	
5	3	3	409	7		4.9	Sample numbers: <b>1010052-4,5,6</b>	
7							Date received by lab: <b>4/6/90</b>	
8							Laboratory location: <b>MI 48103</b>	
10							Seal cost: <b>6.00</b>	

Number of Alarms: \_\_\_\_\_ "MICH" Passed?  Yes  No

Field Mobility Throughput (gallons): \_\_\_\_\_

**6. Comments**

Sample #	Date	Time	Analyst	Comments	Code(s)	T	I
4	4/3/90	13:21				2	1
4	4/3/90	13:27				2	2
5	4/3/90	13:33				1	2
5	4/3/90	13:34				2	2

T = Transducer 1, 2, or 3; I = Instrument 1 or 2

Form 100-01 3/8/90

Signature: **Lula Kottler** Date: **4/6/90** Signature: **E. Caldwell** Date: **4/6/90**

Person Receiving Copy of Form: \_\_\_\_\_ Inspector's Name (print): **Bill Blalock** Inspector's Signature: **Bill Blalock**

SEPA Form 1000-5 (Rev. 3/89) Previous versions are obsolete



## Attachment F

## Target Pressure Rise of Clean and Dry Chambers After Heating to 100°F

Pre-assembly Chamber Temperature ° F	Pressure - psi	
	Minimum	Maximum
62	0.84	1.14
63	0.91	1.11
64	0.88	1.08
66	0.88	1.05
66	0.82	1.02
67	0.78	0.99
68	0.77	0.97
69	0.74	0.94
70	0.71	0.91
71	0.68	0.88
72	0.68	0.85
73	0.62	0.82
74	0.59	0.79
75	0.57	0.77
76	0.54	0.74
77	0.51	0.71
78	0.48	0.68
79	0.46	0.66
80	0.43	0.63
81	0.40	0.60
82	0.37	0.57
83	0.35	0.55
84	0.32	0.52
85	0.29	0.49
86	0.27	0.47
87	0.24	0.44
88	0.21	0.41
89	0.19	0.39
90	0.16	0.36
91	0.13	0.33
92	0.11	0.31
93	0.08	0.28
94	0.05	0.25
95	0.03	0.23

Table based on the ideal gas law and trial tests at a barometer reading of 29.07 in Hg

Page 1  
08/27/90

Attachment G

**Analysis of Laboratory Duplicates**

Precision Data for the Analysis of RVP of Gasoline

Sample Id	Date of Analysis	Vapor Pressure - psi			Within the 95% Confidence Interval
		Initial	Duplicate	Difference	
1008637-1 Sample was cloudy	08/27/90	8.66	8.62	-0.16	Yes
1011005-1	08/07/90	9.71	9.85	0.14	Yes
1011007-1	08/13/90	8.31	8.31	0.00	Yes
1011018-1	08/13/90	9.51	9.63	0.02	Yes
1011037-1	08/13/90	8.89	8.80	-0.09	Yes
1011049-1	08/13/90	8.30	8.36	0.06	Yes
1011055-1	08/14/90	9.58	9.63	0.05	Yes
1011081-1 Sample was cloudy	08/14/90	8.37	8.42	0.05	Yes
1011084-3	08/14/90	10.31	10.29	-0.02	Yes
1011079-1 Sample was cloudy	08/21/90	8.80	8.70	-0.10	Yes
1011082-1	08/21/90	8.85	8.73	-0.08	Yes
1011083-1 Sample was cloudy	08/21/90	8.89	8.82	-0.08	Yes
1011093-1 Sample was cloudy	08/21/90	8.74	8.87	0.07	Yes
1011101-4	08/22/90	10.41	10.41	0.00	Yes
1011102-2	08/22/90	10.42	10.42	0.00	Yes
1011105-4	08/22/90	11.14	11.20	0.06	Yes
1011106-6	08/22/90	10.97	10.91	-0.06	Yes
1011105-6	08/22/90	10.73	10.69	-0.14	Yes
1011109-6	08/22/90	10.99	10.97	-0.02	Yes
1011134-1	08/08/90	10.45	10.45	0.00	Yes
1011134-2	08/08/90	10.27	10.24	-0.03	Yes
1011255-6	08/01/90	10.40	10.27	-0.13	Yes
1011268-2 Sample was cloudy	08/15/90	10.75	10.79	0.04	Yes
1011295-3 Sample was cloudy	08/15/90	10.69	10.76	0.07	Yes
1011289-11 Sample was cloudy	08/15/90	10.68	10.69	0.06	Yes
1011290-12 Sample was cloudy	08/15/90	10.80	10.84	0.04	Yes
1011289-1 Sample was cloudy	08/15/90	10.38	10.48	0.08	Yes
1011289-3 Sample was cloudy	08/15/90	10.39	10.46	0.06	Yes

95 % Confidence Interval: -0.22 &lt;= (Duplicate-Initial) &lt;= 0.20

Comments (when applicable) follow the sample

### Analysis of Laboratory Standards

Data for the Analysis of RVP Standards

Sample Id	Date of Analysis	Vapor Pressure - psi			Within the 95% Confidence Interval
		Average	Measured	Difference	
2,2-dimethylbutane	08/02/90	9.88	9.95	0.08	Yes
2,2-dimethylbutane	08/02/90	9.89	9.78	-0.13	Yes
2,2-dimethylbutane	08/06/90	9.88	9.87	-0.02	Yes
2,2-dimethylbutane	08/08/90	9.89	9.98	0.09	Yes
2,2-dimethylbutane	08/14/90	9.89	9.86	-0.03	Yes
2,2-dimethylbutane	08/14/90	9.89	9.88	-0.03	Yes
2,2-dimethylbutane	08/16/90	9.89	9.90	0.01	Yes
2,2-dimethylbutane	08/20/90	9.88	9.94	0.05	Yes
2,2-dimethylbutane	08/20/90	9.89	10.03	0.14	Yes
2,2-dimethylbutane	08/24/90	9.89	9.88	0.09	Yes
2,2-dimethylbutane	08/24/90	9.89	9.86	-0.03	Yes
2,2-dimethylbutane	08/30/90	9.88	9.95	0.08	Yes
3-methylpentane	08/01/90	6.23	6.39	0.16	Yes
3-methylpentane	08/01/90	6.23	6.38	0.13	Yes
3-methylpentane	08/07/90	6.23	6.41	0.18	Yes
3-methylpentane	08/07/90	6.23	6.25	0.02	Yes
3-methylpentane	08/13/90	6.23	6.08	-0.16	Yes
3-methylpentane	08/13/90	6.23	6.14	-0.09	Yes
3-methylpentane	08/23/90	6.23	6.37	0.14	Yes
3-methylpentane	08/23/90	6.23	6.33	0.10	Yes
3-methylpentane	08/23/90	6.23	6.25	0.02	Yes
3-methylpentane	08/29/90	6.23	6.27	0.04	Yes
3-methylpentane	08/29/90	6.23	6.22	-0.01	Yes
3-methylpentane	08/29/90	6.23	6.27	0.04	Yes
3-methylpentane	08/29/90	6.23	6.22	-0.01	Yes
iso-octane	08/06/90	2.02	1.98	-0.03	Yes
iso-octane	08/10/90	2.02	1.99	-0.03	Yes
iso-octane	08/10/90	2.02	1.95	-0.07	Yes
iso-octane	08/16/90	2.02	2.10	0.08	Yes
iso-octane	08/18/90	2.02	1.99	-0.03	Yes
iso-octane	08/22/90	2.02	2.02	0.00	Yes
iso-octane	08/22/90	2.02	1.98	-0.04	Yes
iso-octane	08/28/90	2.02	2.04	0.02	Yes

95 % Confidence Interval of Vapor Pressure Standards in psi

Standard	Lower	Upper
iso-octane	1.84	2.20
3-methylpentane	6.05	6.42
2,2-dimethylbutane	8.70	10.08
n-pentane	15.18	15.89

## Attachment I

**Statistical Summary  
Vapor Pressure of Gasoline**

	ASTM D 4955 Guidelines	EOD pre-enforcement	1989 Enforcement	May 1990	June 1990	
<b>Precision</b>						
<b>Repeatability</b>						
Field Duplicates	n	270	696	(a)	6	10
95 % Confidence Interval		±0.25	±0.24	(a)	±0.14	±0.11
Lab Duplicates	n	(a)	49	257	93	113
Bias		(a)	-0.04	-0.01	0.00	0.00
Upper 95 % Confidence Interval		(a)	0.15	0.20	0.19	0.17
Lower 95 % Confidence Interval		(a)	-0.23	-0.22	-0.18	-0.17
Reproducibility	n	270	(a)	37	15	11
95 % Confidence Interval		±1.00	(a)	±0.78	±0.84	±0.31
<b>Accuracy</b>						
Iso-octane	n	(a)	9	(a)	4	11
Average Measured Pressure		(a)	2.02	(a)	1.98	2.01
Upper 95 % Confidence Interval		(a)	2.20	(a)	2.14	2.23
Lower 95 % Confidence Interval		(a)	1.84	(a)	1.81	1.78
3-methylpentane	n	(a)	6	19	6	7
Average Measured Pressure		(a)	6.29	6.23	6.29	6.19
Upper 95 % Confidence Interval		(a)	6.55	6.42	6.46	6.25
Lower 95 % Confidence Interval		(a)	6.04	6.05	6.13	6.02
2,2-dimethylbutane	n	(a)	45	26	9	11
Average Measured Pressure		(a)	9.92	9.89	9.95	9.89
Upper 95 % Confidence Interval		(a)	10.14	10.08	10.12	10.09
Lower 95 % Confidence Interval		(a)	9.69	9.70	9.77	9.68
3,3-dimethylbutane-1	n	(a)	11	19	(a)	(a)
Average Measured Pressure		(a)	13.07	13.01	(a)	(a)
Upper 95 % Confidence Interval		(a)	13.31	13.17	(a)	(a)
Lower 95 % Confidence Interval		(a)	12.82	12.84	(a)	(a)
n-pentane	n	(a)	37	20	6	9
Average Measured Pressure		(a)	15.43	15.38	15.38	15.38
Upper 95 % Confidence Interval		(a)	15.61	15.60	15.51	15.54
Lower 95 % Confidence Interval		(a)	15.25	15.16	15.25	15.22
Method Detection Limit		(a)	(a)	(a)	0.250	(a)

(a) not addressed

Attachment J

### Precision and Accuracy Calculation Formulas Vapor Pressure of Gasoline

Statistic	Formula
<b>Repeatability field duplicates</b> difference Standard Deviation confidence limits around an individual analysis confidence limits around the difference between two analyses of the same materials within the same laboratory	$d = \text{absolute}(\text{duplicate} - \text{initial})$ $s_x = (\sum d^2 / 2n)^{1/2}$ $= \pm (t_{n, 0.975})^* (s_x)$  $= \pm (2^{1/2})^* (t_{n, 0.975})^* (s_x)$
<b>Repeatability lab duplicates</b> difference average bias Standard Deviation confidence limits around the average bias for the difference between the original-sample and the second-sample value from the same container	$d = (\text{duplicate} - \text{initial})$ $\bar{d} = \sum d / n$ $s_d = ((\sum d^2 - (\sum d)^2 / n) / (n - 1))^{1/2}$  $= d_x \pm (t_{n-1, 0.975})^* (s_d)$
<b>Reproducibility</b> difference Standard Deviation confidence around an individual observation confidence interval for the difference between a single test result obtained in one laboratory and a single test result obtained in another laboratory on the same material	$d = \text{absolute}(\text{duplicate} - \text{initial})$ $s_x = (\sum d^2 / 2n)^{1/2}$  $= \pm (t_{n, 0.975})^* (s_x)$  $= \pm (2^{1/2})^* (t_{n, 0.975})^* (s_x)$
<b>Accuracy</b> difference average bias Standard Deviation confidence interval around the average bias for an individual analysis	$d = (\text{observed} - \text{average})$ $\bar{d} = \sum d / n$ $s_d = ((\sum d^2 - (\sum d)^2 / n) / (n - 1))^{1/2}$  $= d_x \pm (t_{n-1, 0.975})^* (s_d)$
<b>Method Detection Limit</b> Standard Deviation, $s_x$ , where x is a standard at a concentration very close to or at the method detection limit Method Detection Limit (one-sided 99% confidence limit on an individual observation)	$s_x = ((\sum x^2 - (\sum x)^2 / n) / (n - 1))^{1/2}$  $= (t_{n-1, 0.99})^* (s_x)$